

Fig. 1a

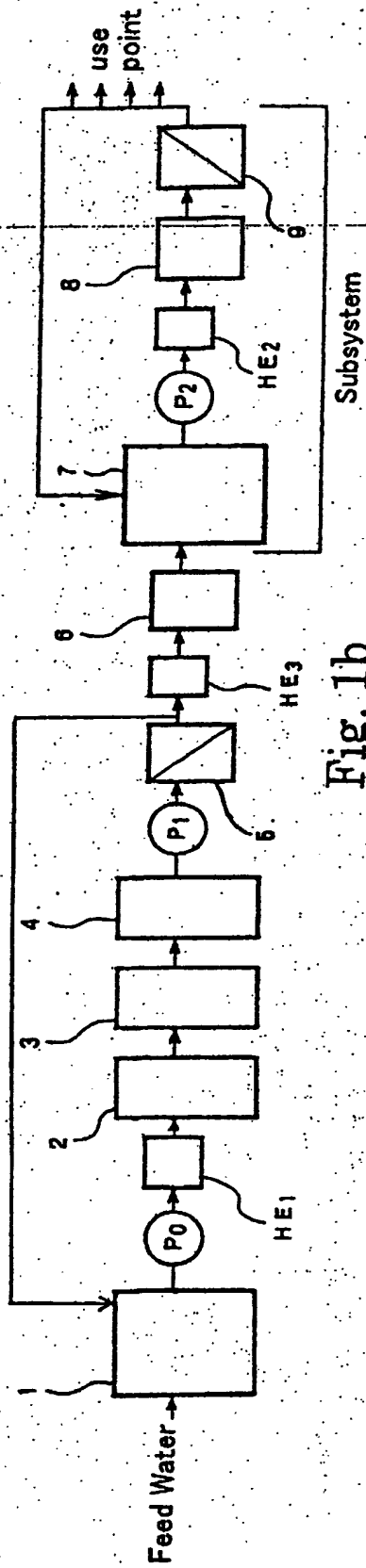


Fig. 1b

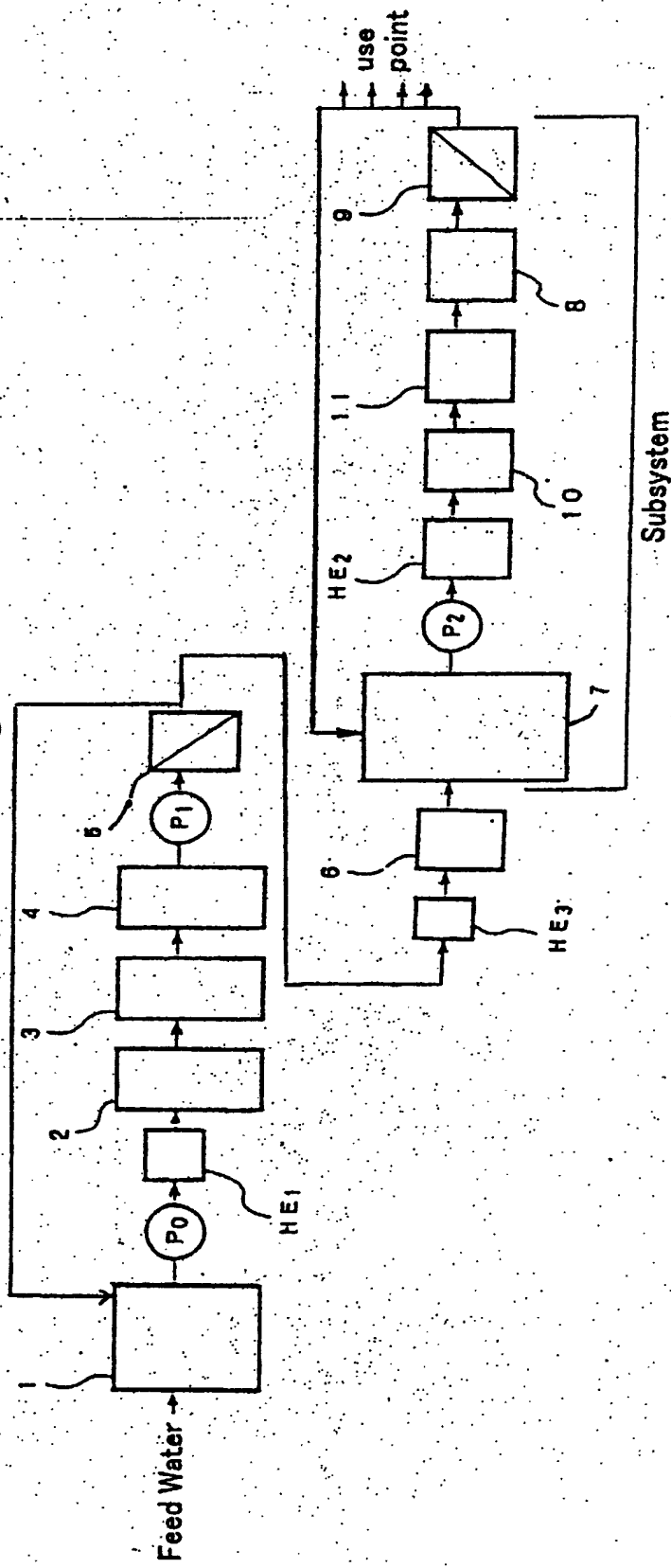


Fig. 2a

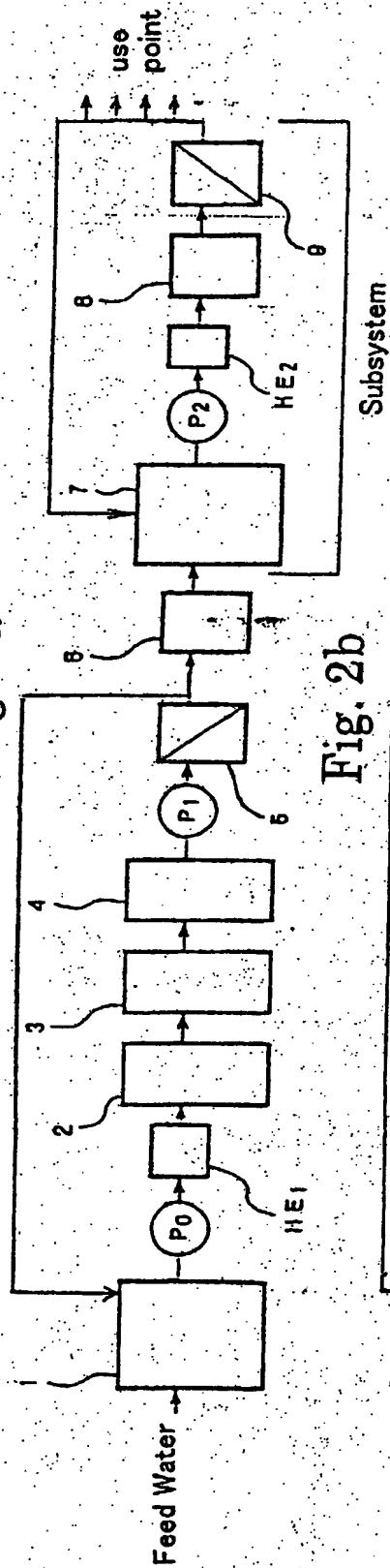


Fig. 2b

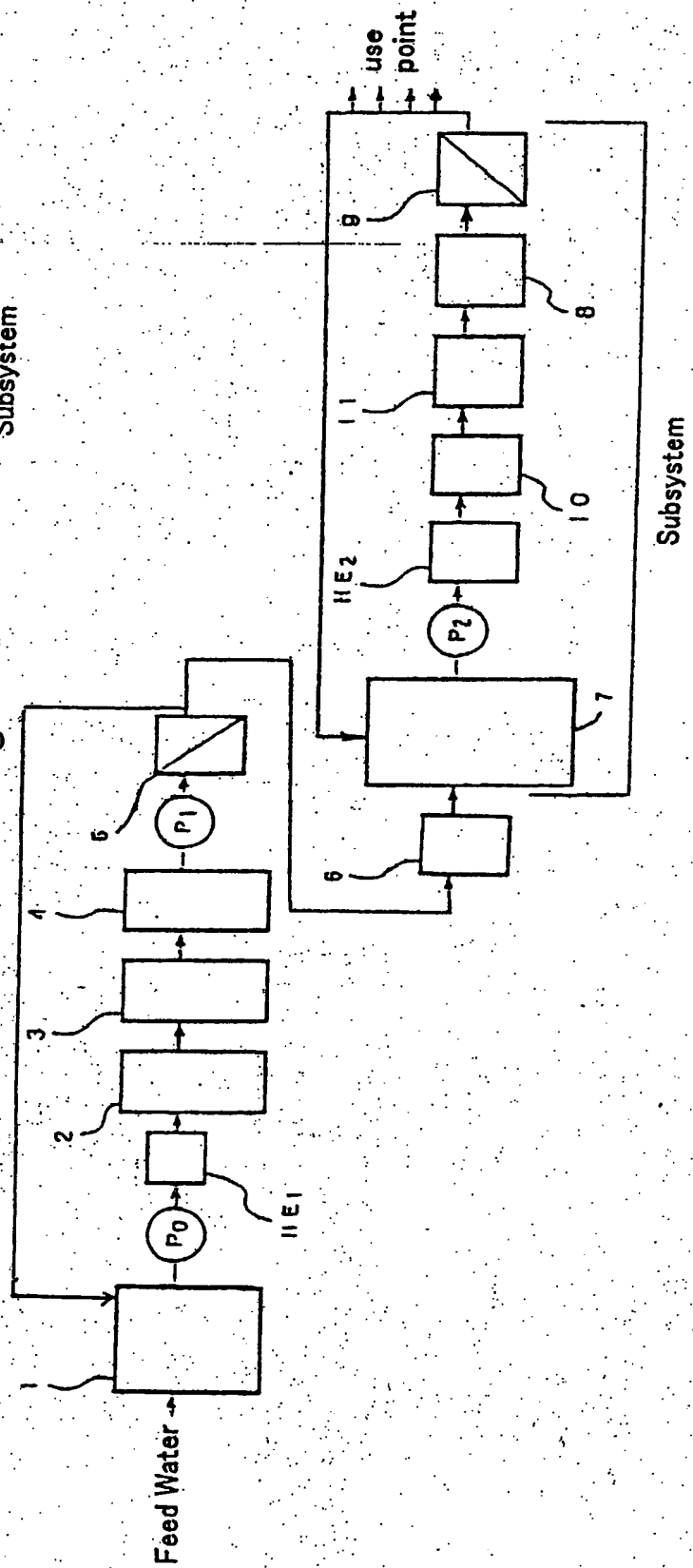
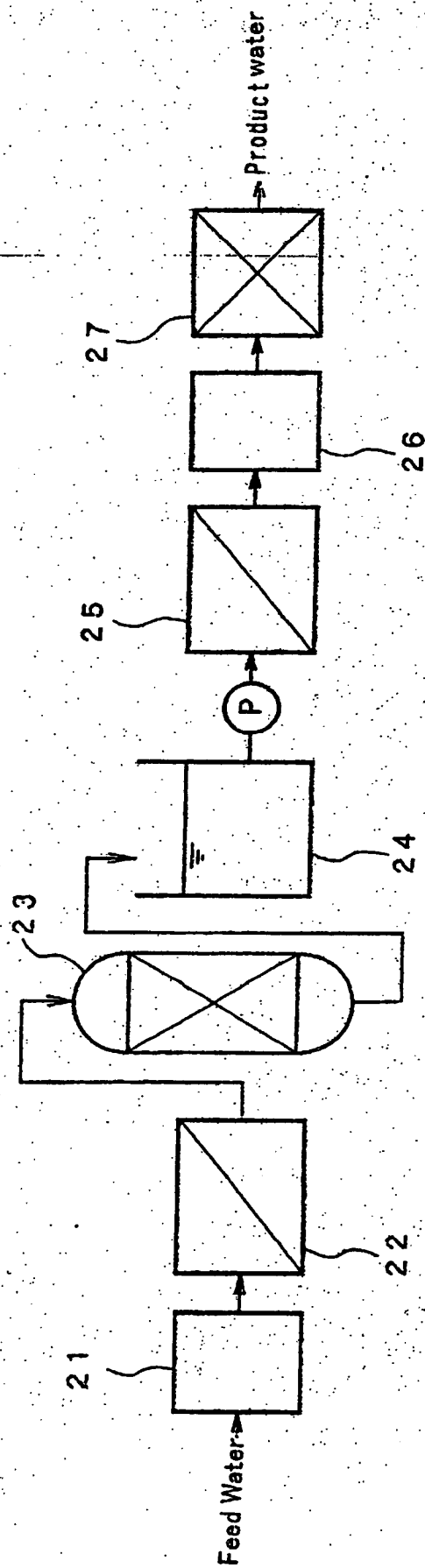
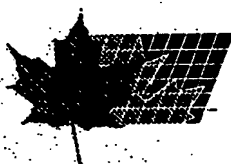


Fig. 3





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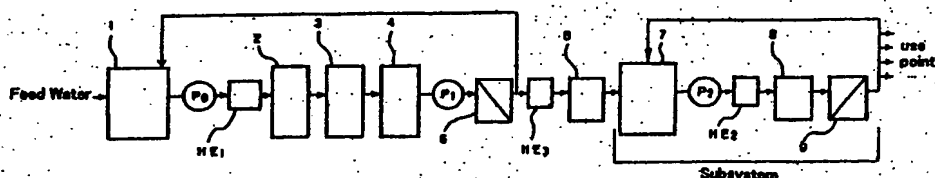
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(54) **METHODE POUR DESINFECTER UN APPAREIL DE  
DESIONISATION DE L'EAU ET METHODE POUR PRODUIRE  
DE L'EAU DESIONISEE**

(54) **METHOD OF DISINFECTING A DEIONIZED WATER  
PRODUCING APPARATUS AND METHOD OF PRODUCING  
DEIONIZED WATER**



(57) An electrodeionization apparatus for deionized water is prevented from bacteria proliferation therein. The deionized water producing apparatus and the electrodeionization apparatus filled with ion exchangers in the diluting compartment thereof. Hot water of more than 80°C is fed into the RO apparatus and hot water of more than 60°C is fed to the electrodeionization apparatus.



# Method of disinfecting a deionized water producing apparatus and method of producing deionized water

## Field of the invention

The present invention relates to a method of disinfecting deionized water producing apparatus and a method of producing deionized water with using the electrodeionization apparatus disinfected by disinfecting method.

## 10 Background of the invention

A conventional system for producing purified water which is employed in fields of the pharmaceutical manufacturing industry, the semiconductor manufacturing industry and the like is shown Figs. 2a and 2b.

Fig. 2a is a system diagram illustrating a system for producing deionized water employed in the field of the pharmaceutical manufacturing industry, in which raw water is treated with an activated carbon (AC) column or tower 2, a safety filter 3 and a membrane degassing apparatus 4 by way of a tank 1, a pump  $P_0$  and a heat exchanger  $HE_1$ , and then the water is  
20 pressured by a pump  $P_1$  and treated with a reverse osmosis

membrane (RO) apparatus 5 and a electrodeionization apparatus 6, after that, the water is treated with a subsystem comprising a ultraviolet (UV) disinfecting apparatus 8 and an ultra filtration (UF) membrane apparatus 9 by way of a tank 7, a pump  $P_2$  and a heat exchanger  $HE_2$ , and finally the water is transported to a use point.

Fig. 2b is a system diagram illustrating a system for producing the deionized water employed in the field of the semiconductor manufacturing industry. As shown in Fig.2b, the raw water such as city water and well water is treated with the AC column 2, the safety filter 3 and the membrane degassing apparatus 4 by way of the tank 1, the pump  $P_0$  and the heat exchanger  $HE$ , and then pressurized by the pump  $P_1$ . After that, the water is treated with the RO membrane apparatus 5 and the electrodeionization apparatus 6.

Furthermore, the water treated with a subsystem comprising a low - pressure UV oxidizing apparatus 10, a mixed-bed ion exchange apparatus 11, the UV disinfecting apparatus 8 and the UF membrane apparatus 9, and then transported to the use point.

The deionized water producing apparatus is disinfected

when the operation of the apparatus is commenced or periodically as follows.

Above equipments previous to the electrodeionization apparatus 6 are disinfected with heated water or agents. For example, when the system shown in Fig.2a is disinfected with the heated water, the water in the tank 1 is heated to 80 to 90°C with the heat exchanger HE<sub>1</sub>, and then the hot water is let through the AC column 2, the safety filter 3 and the membrane degassing apparatus 4, after that, the water is pressurized with pump P<sub>1</sub> to let the water through the RO membrane apparatus 5. The reject water of the RO membrane apparatus is drained or fed back to the tank 1. The permeated water of the RO membrane apparatus is drained or circulated to the tank 1.

After that, the subsystem is disinfected with heated water or agents. For example, disinfection is made in such a manner that the water in the tank 7 is heated to 80 to 90°C with the heat exchanger HE<sub>2</sub> and, then, is let through the UV disinfecting apparatus 8 and the UF membrane apparatus. The reject water and the permeated water of the UF membrane apparatus 9 are drained. Furthermore, piping from the usepoint to the tank 7 is disinfected with steam.

The electrodeionization apparatus 6 is disinfected by using a germicide such as agent like  $H_2O_2$ . However, the efficiency of disinfection is insufficient. In case the disinfecting with agents is periodically made, it is necessary to securely manage the prevention against retention of the agents in the equipments and the piping.

As disclosed above, the conventional systems for producing deionized water shown in Figs. 2a and 2b is disinfected insufficiently and bacteria are present in product water of the electrodeionization apparatus, so that the succeeding subsystem is polluted with the bacteria within a short time. Since the bacteria are not completely removed even with the UV disinfecting apparatus, the bacteria proliferate in the system as the time elapses. That is, an ion exchanger resin or an ion exchange membrane is disinfected insufficiently, so that the bacteria are unusually present on the order of  $10^2$  to  $10^7$  per 100cc in the product water. The number of bacteria increases as the operational time elapses to degrade the quality of the product water.

Object and summary of the invention



It is an object of the present invention to solve  
aforementioned conventional problems and to provide a method of  
disinfecting deionized water producing apparatus and a method of  
producing deionized water which prevents bacteria proliferation in  
the electrodeionization and provides deionized water of high  
quality.

10 A method of the present invention is for disinfecting  
deionized water producing apparatus which has a preceding  
apparatus including a reverse osmosis (RO) apparatus, and an  
electrodeionization apparatus having a diluting compartment  
filled with an ion exchanger. According to the method, the  
preceding apparatus is disinfected by flowing hot water of higher  
than 80°C therethrough, and the electrodeionization apparatus is  
disinfected by flowing hot water of higher than 60°C therethrough.

20 The deionized water producing apparatus may be  
disinfected by the method of the present invention when the  
apparatus is started to be operated or intermittently during  
operation thereof, so that the number of bacteria in the deionized  
water flown out of the electrodeionization apparatus is kept at a  
low level.

The hot water to be flown into the apparatus for disinfection

thereof is preferably heated or cooled at a rate of  $0.1 - 10^{\circ}\text{C}/\text{min}$  in order to prevent deterioration by heat of ion exchange resin and ion exchanging membranes in the electrodeionization apparatus.

According a method of producing deionized water of the present invention, deionized water is prepared by the deionized water producing apparatus which is disinfected according to the disinfecting method of the present invention.

#### Brief description of the drawings

10        Figs. 1a and 1b show an electrodeionization apparatus which is used for a method of producing deionized water of the present invention;

Fig. 2 is a flow diagram of a conventional electrodeionization apparatus; and

Fig. 3 is a flow diagram of an electrodeionization apparatus employed by an example of the invention.

#### Detailed description of the preferred embodiments

20        The invention will be described referring to the attached drawings. Figs. 1a and 1b show an electrodeionization apparatus employed by the method of the invention, where the same

reference numerals denote the same member as in Figs. 2a and 2b.

The apparatus of Figs. 1a and 1b has the same construction as of Figs. 2a and 2b except that the heat exchanger  $HE_3$  is provided preceding the electrodeionization apparatus 6.

The preceding apparatus including the RO apparatus is disinfected by flowing hot water of higher than  $80^{\circ}\text{C}$  therethrough, and the electrodeionization apparatus is disinfected by flowing hot water of higher than  $60^{\circ}\text{C}$  therethrough. After the apparatuses and members preceding the electrodeionization apparatus is  
10 disinfected by the hot water of higher than  $80^{\circ}\text{C}$ , water of ambient temperature is flown through the preceding apparatus, and the treated water flown out of the preceding apparatus is heated up to  $60^{\circ}\text{C}$  or higher and fed to the electrodeionization apparatus for disinfecting thereof.

For example in Figs. 1a and 1b, raw water in the tank 1 is heated by the heat exchanger  $HE_1$  up to  $80^{\circ}\text{C}$  or higher, preferably 80 to  $90^{\circ}\text{C}$  and the heated hot water is flown through the RO apparatus 5 for disinfection thereof.

After disinfecting the RO apparatus 5, water of ambient  
20 temperature in the tank 1 is fed to the preceding apparatus and permeated water taken out of the RO apparatus 5 is heated up to

60°C or higher to be fed to the electrodeionization apparatus 6.

The apparatus 6 has ion exchangers including cation exchange resin and anion exchange resin, cation exchange membranes, anion exchange membranes, an anode plate and a cathode plate, and all of which are disinfected by the hot water.

The hot water disinfecting the electrodeionization apparatus is preferable to have equal to or higher quality than that of permeated water of a RO apparatus.

Accordingly in the present invention, after the preceding apparatus including the RO apparatus is first disinfected by the hot water, water of ambient temperature is flown therethrough, and the permeated water is heated and fed to the electrodeionization apparatus for disinfection. The hot water for disinfecting the electrodeionization apparatus should be 60°C or higher, preferably higher than 70°C and more preferably higher than 80°C.

The hot water is preferable to flow at a rate of 0.25 - 1.00L/min/cell, where "L" is liter, for more than 10 minutes, more preferably more than 15 minutes, and most preferably more than 30 minutes.

When the hot water of 60°C is flown, it is preferable to be

flowed for more than 20 minutes, more preferably more than 40 minutes, and most preferably more than 60 minutes.

When the hot water of 80°C is flown, it is preferable to be flown for more than 10 minutes, more preferably more than 20 minutes, and most preferably more than 40 minutes.

The water fed to the electrodeionization apparatus is unpreferable to be charged from hot to cool or from cool to hot suddenly, since it heats or cools ion exchange membranes to expand or retract so rapidly to cause deterioration thereof.

10 Accordingly, the water fed to the electrodeionization apparatus is preferable to be heated up or cooled down at a rate of 0.1 - 10°C /min.

After disinfecting the electrodeionization apparatus by the hot water, then the temperature of the hot water fed to the electrodeionization is fallen gradually at a rate of 0.1 - 10°C down to 40°C or lower, preferably 35°C or lower, most preferably to the ambient temperature.

20 The flown out water of the electrodeionization apparatus which is fed with the hot water may be discarded or returned to the tank 1. Concentrated water flown out of a concentrating compartment of the electrodeionization apparatus may be also

discarded or returned to the tank 1.

The water pressure at each inlet of the diluting compartment, the concentrating compartment and the electrode compartment arranged in the electrodeionization apparatus is preferably less than 0.1MPa and more preferably less than 0.05MPa in order to prevent deformation or deterioration of the apparatus.

A member of the apparatus such as a pipe or the tank which contacts the hot water is preferably of heat resistant material such as stainless.

Although the apparatus shown in Figs. 1a and 1b relates to the purified water producing system for the pharmaceutical manufacturing industry and ultra - pure water producing system for the semiconductor manufacturing industry, the method of the present invention is not limitative thereto but capable of being applied to other various fields.

## Examples

### Example 1

City water of Atsugi, Kanagawa Japan was treated at a rate of 0.5m<sup>3</sup>/hr by the apparatus of Fig.3.

In this apparatus, feed water of the above city water was flown through the heat exchanger 21, treated by the microfiltration (MF) apparatus 22 and the activated carbon (AC) tower 23, and fed from the tank 24 to the RO apparatus 25 via the pump P. Permeated water from the RO apparatus 25 was heat-exchanged by the heat exchanger 26 and treated by the electrodeionization apparatus 27. The particulars of the apparatuses are as follows:

MF apparatus 22: "Kuriequrun" of Kurita Water Industries Ltd.

AC tower 23: "Kurare Coal KW" of Kurare Co., Ltd.

RO apparatus 27: "SG 4040CZH of Desari Co., Ltd. having a diameter of 4 inches

Electrodeionization apparatus 27: "CDI P - 10 of U.S. Filter / Ionpure, Inc. having 10 cells; mixture of anion exchanger and cation exchanger being filled in both diluting compartment and concentrating compartment:

The deionized water apparatus 27 is of the type where feed water at an ambient temperature is flown at a rate of 0.83L/min/cell.

First step;

In the first step, hot water of 80°C was flown from the heat exchanger to the RO apparatus via the apparatus 22, the tower 23, the tank 24 and the pump so that they were disinfected.

Second step:

After the first step, water of ambient temperature was flown through the heat exchanger, the apparatus 22, the tower 23, the tank 24, the pump P, the apparatus 25, the heat exchanger 26 and the electrodeionization 27. The inlet pressure of the apparatus 27 was adjusted to 0.05Mpa. Then the water was  
10 heated by the heat exchanger 26 at a rate of 1-1.5°C/min for about 40 minutes such that the temperature of the water flown out from the diluting compartment became at 60°C. The hot water was flown through the electrodeionization apparatus 27 for one hour, and thereafeter the hot water was started to be cooled down at a rate of 1-1.5°C/min. The hot water was cooled down until the temperature at the outlet of the diluting compartment became to 35°C. The electrodeionization apparatus 27 was not flown with electric current during the second step.

Third step:

20 After the second step, the raw water was fed to the apparatus of Fig. 3, and the permeated water of the RO apparatus



25 was fed to the electrodeionization apparatus 27 continuously for 9 days. The number of bacteria in the permeated water of the RO apparatus 25 and the deionized water flown out of the electrodeionization 27 was measured, and the results thereof are shown in Table 1.

The uppermost line in Table 1 shows the number of bacteria in the water before disinfecting the apparatus.

Table 1.

Days after disinfection	number of bacteria per 1cc in the permeated water	number of bacteria per 1cc in the deionized water
before disinfection	75	300
after 1 day	0.00	0.06
after 3 days	0.00	0.11
after 6 days	0.06	not detected
after 9 days	0.01	0.05

As clearly shown in Table 1, the number of bacteria is kept very low for a long period when the apparatus such as the MF apparatus and the RO apparatus preceding the electrodeionization is disinfected with hot water of 80°C and the electrodeionization is disinfected with hot water of 60°C.

As described above, the electrodeionization apparatus is prevented from bacteria proliferating therein so that the number

of bacteria flowing out of the electrodeionization apparatus is kept low, according to the present invention.

Accordingly a UV disinfecting apparatus succeeding the electrodeionization apparatus can be deleted, and times of disinfecting the subsystem are reduced, so that deionized water of high quality is produced at a very low cost.

What is claimed is:

1. Method of disinfecting a deionized water producing apparatus which comprises a preceding apparatus including a reverse osmosis apparatus, and an electrodeionization apparatus having a diluting compartment filled with an ion exchanger,

wherein the preceding apparatus is disinfected by flowing hot water of higher than 80°C therethrough, and

wherein the electrodeionization apparatus is disinfected by flowing hot water of higher than 60°C therethrough.

10 2. The method of disinfecting a deionized water producing apparatus as claimed in claim 1, wherein at first the preceding apparatus is disinfected by flowing hot water of higher than 80°C therethrough, then water of ambient temperature is flown through the preceding apparatus, and the treated water flowing out of the preceding apparatus is heated and flown through the electrodeionization apparatus for disinfecting thereof.

3. The method of disinfecting a deionized water producing apparatus as claimed in claim 1, wherein said hot water is flown through the electrodeionization apparatus at a rate of 0.25-1.00  
20 L/min/cell for more than 10 minutes.

4. The method of disinfecting a deionized water producing

apparatus as claimed in claim 1, wherein the hot water flown thorough the electrodeionization apparatus is heated at a rate of 0.1-10°C/min.

5. The method of disinfecting a deionized water producing apparatus as claimed in claim 1, wherein the hot water flown into the electrodeionization is reduced its temperature at a rate of 0.1-10°C/min after disinfecting the electrodeionization.

6. Method of producing deionized water with using the deionized water producing apparatus disinfected according to the method of claim 1.

10

### Abstract of the disclosure

An electrodeionization apparatus for deionized water is prevented from bacteria proliferation therein. The deionized water producing apparatus and the electrodeionization apparatus filled with ion exchangers in the diluting compartment thereof. Hot water of more than 80°C is fed into the RO apparatus and hot water of more than 60°C is fed to the electrodeionization apparatus.